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Synthesis and structures of three isoxazole-containing Schiff bases

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The synthesis and structures of three isoxazole-containing Schiff bases are reported, namely, (*E*)-2-{[(isoxazol-3-yl)imino]methyl}phenol, $C_{10}H_8N_2O_2$, (*E*)-2-{[(5-methylisoxazol-3-yl)imino]methyl}phenol, $C_{11}H_{10}N_2O_2$, and (*E*)-2,4-di-*tert*-butyl-6-{[(isoxazol-3-yl)imino]methyl}phenol, $C_{18}H_{24}N_2O_2$. All three structures contain an intramolecular O—H···N hydrogen bond, alongside weaker intermolecular C—H···N and C—H···O contacts. The C—O(H) and imine C=N bond lengths were consistent with structures existing in the enol rather than the keto form. Despite having dihedral angles <25°, none of the compounds were observed to be strongly thermochromic, unlike their anil counterparts; however, all three compounds showed a visible colour change upon irradiation with UV light.

1. Introduction

A wide range of Schiff bases can be relatively easily prepared making them versatile as ligands and consequently they have found widespread use over many years in areas such as organometallic chemistry (Kargar *et al.*, 2020), polymer synthesis (Mighani, 2020), anticancer drugs (Parveen, 2020), catalysts (Kumari *et al.*, 2019) and sensors (Sahu *et al.*, 2020). In addition, Schiff bases themselves have been found to display interesting properties with anils, *i.e.* Schiff bases of salicylaldehyde derivatives with aniline derivatives, having been first found to exhibit both thermo- and photochromism in the solid state (Senier *et al.*, 1909; Cohen & Schmidt, 1962; Cohen *et al.*, 1964). Originally, the thermo- and photochromism of anils were thought to be mutually exclusive (Cohen & Schmidt, 1962; Cohen *et al.*, 1964), but this has since been found not to be the case and it is thought they all display thermochromism with some also displaying photochromism (Fujiwara *et al.*, 2004). The colour change is believed to be due to a photo- or thermally induced tautomeric equilibrium shift between colourless enol(–imine) and keto(–amine) forms (Hadjoudis & Mavridis, 2004; Robert *et al.*, 2009).

The Schiff bases of salicylaldehyde (2-hydroxybenzaldehyde) derivatives with isoxazole derivatives have not been widely characterized structurally, with a search of the Cambridge Structural Database (CSD; Version of June 2020; Groom *et al.*, 2016) revealing two structures, namely, (*E*)-2-methoxy-6-{[(5-methylisoxazol-3-yl)imino]methyl}phenol (refcode GITGIA; Zhao *et al.*, 2008) and *N*-(5-methylisoxazol-3-yl)-3,5-di-*tert*-butylsalicylaldimine (refcode YINFAD; Çelik *et al.*, 2007). Herein the synthesis and characterization of three isoxazole-containing Schiff bases are reported, namely, (*E*)-2-{[(isoxazol-3-yl)imino]methyl}phenol, **1**, (*E*)-2-{[(5-methylisoxazol-3-yl)imino]methyl}phenol, **2**, and (*E*)-2,4-di-*tert*-butyl-6-{[(isoxazol-3-yl)imino]methyl}phenol, **3** (see Scheme 1).

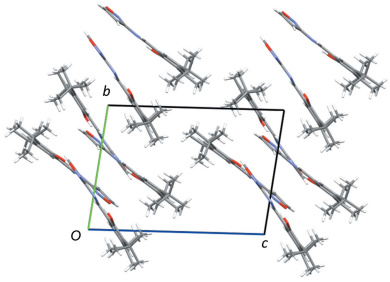


Table 1
Experimental details.

For all structures: Z = 4. Experiments were carried out with Mo $K\alpha$ radiation. H atoms were treated by a mixture of independent and constrained refinement.

	1	**2**	**3**
Crystal data			
Chemical formula	$C_{10}H_8N_2O_2$	$C_{11}H_{10}N_2O_2$	$C_{18}H_{24}N_2O_2$
M_r	188.18	202.21	300.39
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Orthorhombic, $Pna2_1$	Triclinic, $P\overline{1}$
Temperature (K)	210	120	120
a, b, c (Å)	4.5999 (5), 10.2684 (10), 18.711 (2)	20.5584 (7), 10.0468 (4), 4.6417 (2)	10.8955 (5), 10.9571 (4), 14.8329 (6)
α, β, γ (°)	90, 90, 90	90, 90, 90	82.335 (3), 88.326 (4), 75.178 (3)
V (Å^3)	883.79 (16)	958.73 (7)	1696.56 (12)
μ (mm^{-1})	0.10	0.10	0.08
Crystal size (mm)	0.3 × 0.08 × 0.05	0.49 × 0.24 × 0.09	0.6 × 0.31 × 0.18
Data collection			
Diffractometer	Bruker SMART APEXII area detector	Oxford Diffraction Xcalibur (Sapphire3, Gemini ultra)	Oxford Diffraction Xcalibur (Sapphire3, Gemini ultra)
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Analytical (*CrysAlis PRO*; Oxford Diffraction, 2010)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min}, T_{max}	0.654, 0.746	0.969, 0.991	0.833, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10497, 2166, 1978	6756, 2021, 1819	14901, 6942, 5078
R_{int}	0.020	0.040	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667	0.641	0.625
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.083, 1.08	0.037, 0.081, 1.05	0.049, 0.119, 1.02
No. of reflections	2166	2021	6942
No. of parameters	131	141	447
No. of restraints	0	1	0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.14	0.16, −0.17	0.26, −0.22

Computer programs: *APEX2* (Bruker, 2012), *CrysAlis PRO* (Oxford Diffraction, 2010), *SAINT* (Bruker, 2012), *SHELXT2018* (Sheldrick, 2015*a*), *SHELXS97* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

2. Experimental

2.1. Synthesis

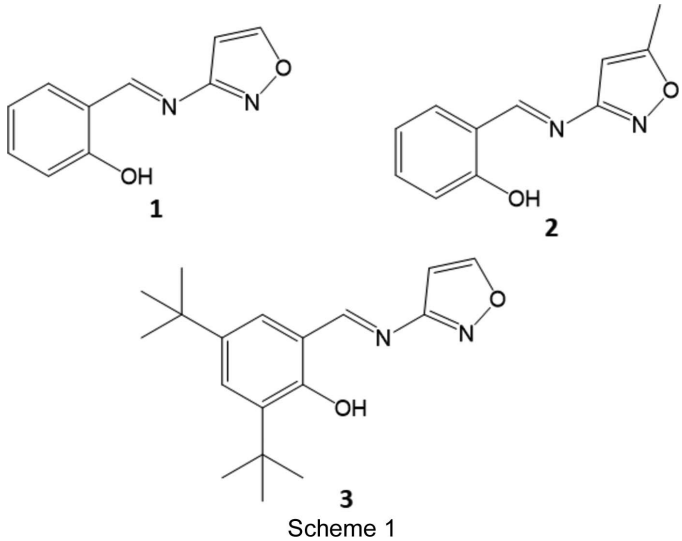
All reagents were used as supplied by Aldrich. Compounds were synthesized by direct condensation of the appropriate salicylaldehyde and isoxazole derivatives in ethanol. The salicylaldehyde (0.0025 mol) and aniline (0.0025 mol) were each dissolved in ethanol (25 ml). The resulting solutions were combined and refluxed with stirring for 6–8 h. Any precipitate was filtered off, rinsed with ethanol and left to dry. The (remaining) solution was then rotary evaporated until (further) precipitate formed. Recrystallization was carried out from hexane–dichloromethane for **1**, ethanol for **2** or chloroform for **3** (see Scheme 1).

2.2. Characterization

Elemental C, H and N content analysis was carried out using the Durham University Analytical service on an Exeter Analytical E-440 Elemental Analyzer. Mass spectrometry in positive electrospray (ES+) mode was performed by the Durham University Mass Spectrometry service on a Waters TQD with an Acquity solvent system. Full details are available in the supporting information.

2.3. Refinement

All H atoms, apart from the hydroxy H atom involved in intramolecular hydrogen bonding with the imine N atom, were positioned geometrically and refined using a riding model. The H atoms involved in the intramolecular hydrogen bonding were located in a Fourier difference map wherever feasible.



Scheme 1

Compounds **1** and **2** crystallized in noncentrosymmetric space groups; however, the Flack parameters obtained were not meaningful as the data were collected with molybdenum radiation and there are no heavy atoms to facilitate anomalous

Table 2
Hydrogen-bond geometry (Å, °) for **1**.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.85 (3)	1.86 (3)	2.6110 (19)	146 (3)
C7—H7···N2^{i}	0.93	2.71	3.599 (2)	159
C9—H9···O1^{ii}	0.93	2.70	3.400 (2)	133
C9—H9···N2^{i}	0.93	2.61	3.403 (2)	144
C10—H10···O1^{i}	0.93	2.52	3.235 (2)	134

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Table 3
Hydrogen-bond geometry (Å, °) for **2**.

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2^{i}	0.95	2.61	3.502 (3)	157
C7—H7···N2^{i}	0.95	2.49	3.394 (3)	159
C9—H9···N2^{i}	0.95	2.74	3.591 (3)	149
C2—H2···O1^{ii}	0.95	2.62	3.496 (3)	153
O1—H1···N1	0.95 (3)	1.80 (3)	2.632 (2)	145 (3)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+2, -y+2, z-\frac{1}{2}$.

dispersion. In **3**, which contained two independent molecules in the asymmetric unit, one of the *tert*-butyl groups was disordered; the sum of the occupancies of the two parts was set to equal 1 and subsequently fixed at the refined values. The interplanar dihedral angle was calculated by measuring the angle between planes computed through the five or six non-H atoms of the two rings. See Table 1 for further details of the crystallographic data collections.

3. Results and discussion

3.1. Structural discussion

The structures of **1**–**3** all consist of the same basic backbone with a hydroxy-substituted arene group joined to an isoxazole ring *via* an imine (C═N) group (Fig. 1). The C7═N1 bond lengths are consistent with the presence of a double bond [ranging from 1.283 (2) Å in **1** to 1.293 (2) Å in **3**], while the C1—O1 bond lengths [ranging from 1.350 (2) Å in **1** to 1.3655 (18) Å in **3**] are consistent with a single bond. Indeed, the hydroxy H atom was located in a Fourier difference map in the vicinity of the O atom, supporting the fact that the structures are all in the more commonly observed enol form rather than the keto form. All three structures contain an intramolecular O1—H1···N1 hydrogen bond with similar parameters, *e.g.* the O1···N1 distances range from 2.6062 (17) to 2.632 (2) Å (Tables 2–4). The structures also contain weaker intermolecular C—H···N and C—H···O interactions (Tables 2–4).

Examining the structure of **1**, short π–π stacking type interactions are found between the six-membered aromatic ring and the C═N group [centroid-to-centroid distance = 3.2905 (3) Å] (Corne *et al.*, 2016), creating one-dimensional stacks in approximately the [101] direction. The intermolecular interactions involving the isoxazole N atom and the OH group are: (i) bifurcated C—H···N interactions to other molecules; (ii) bifurcated C—H···O interactions to two

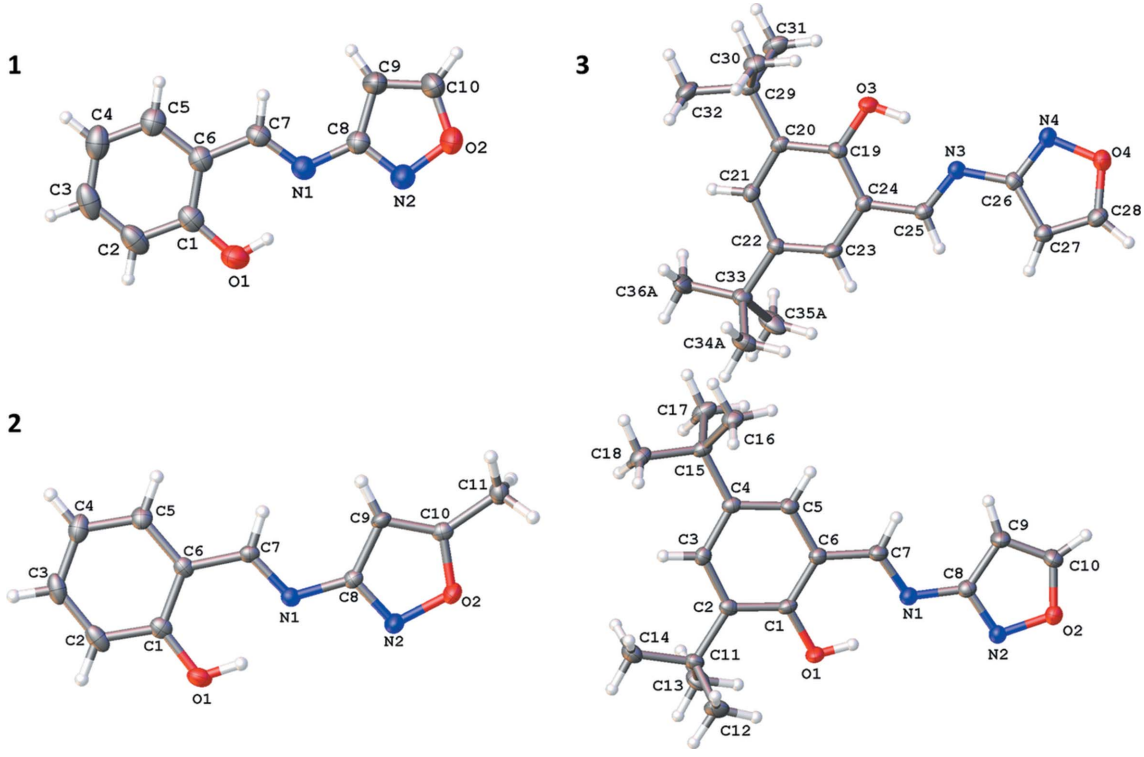


Figure 1
Illustration of the structures of **1** [at 210 (2) K], **2** [120 (2) K] and **3** [120 (2) K], with the atomic numbering schemes depicted. Anisotropic displacement parameters are drawn at the 50% probability level. In the case of **3**, only one position of the disordered *tert*-butyl group is shown for clarity.

Table 4
Hydrogen-bond geometry (Å, °) for **3**.

D—H···A	D—H	H···A	D···A	D—H···A
C23—H23···O2^{i}	0.95	2.60	3.5232 (19)	165
C25—H25···N2^{i}	0.95	2.70	3.637 (2)	169
C5—H5···O4^{ii}	0.95	2.66	3.538 (2)	155
C7—H7···N4^{ii}	0.95	2.82	3.708 (2)	156
C18—H18*B*···N2^{iii}	0.98	2.67	3.559 (2)	152
O1—H1···N1	0.92 (2)	1.76 (2)	2.6207 (18)	153 (2)
O3—H3···N3	0.91 (3)	1.77 (2)	2.6062 (17)	151 (2)
C10—H10···O3^{ii}	0.95	2.53	3.187 (2)	127
C28—H28···O1^{i}	0.95	2.69	3.3370 (19)	126

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x+1, -y+1, -z$; (iii) $-x, -y, -z$.

different molecules. These interactions link a central molecule with four molecules in total, *i.e.* two molecules either side of itself, creating chains in approximately the *b*-axis direction. Combining these interactions with the π–π stacking creates a three-dimensional network with a herringbone-type packing structure (Fig. 2).

The structure of **2** has short π–π stacking type interactions that exist between the six-membered aromatic ring and the C═N group [centroid-to-centroid distance = 3.2772 (1) Å], creating a one-dimensional stack approximately up the [101] direction. All the stacks in the *ac* plane are in the same direction; however, moving in the *b*-axis direction by one molecule, the stacks in the *ac* plane are in different directions due to the presence of the 2_1 screw axes and glide planes. The structure also contains: (i) C—H···N and C—H···O interactions involving the N and O atoms of isoxazole; (ii) C—H···O interactions involving the O atom of the OH group. These interactions link the central molecule to four others, two on each side of the molecule, creating a three-dimensional network. An illustration of the overall packing is shown in Fig. 3.

In **3**, the two independent molecules show slightly different intermolecular interactions: (i) C—H···N (bifurcated for the isoxazole ring containing atoms N2 and O2, and not for the isoxazole ring containing atoms N4 and O4) and a C—H···O interaction involving the N and O atoms of isoxazole; (ii) C—H···O interactions involving the O atom of the OH group. This creates a three-dimensional packing network (Fig. 4).

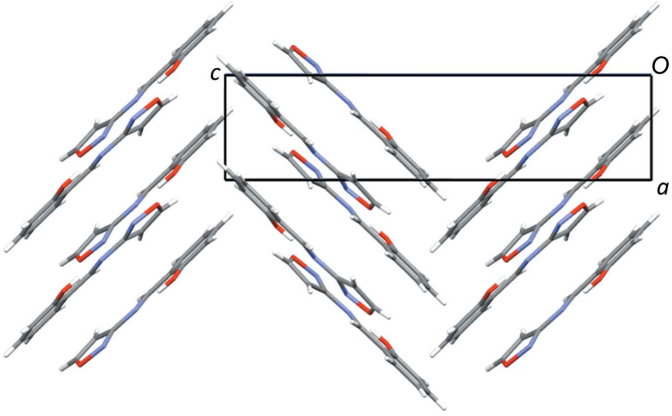


Figure 2
Illustration of the packing in **1**, looking down the *b* axis.

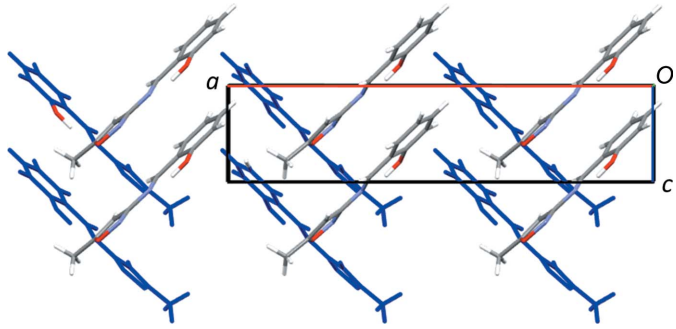


Figure 3
Illustration of the packing in **2**, looking down the *b* axis. Molecules are shown in elemental colours (C grey, O red, N blue and H white) at the front, while molecules shown in blue are one molecule down the *b* axis, showing the different orientations.

There are no π–π stacking type interactions between the six-membered aromatic ring and the C═N group in this case, presumably because of the presence of the bulky *tert*-butyl groups.

3.2. Chromic studies

The chromic behaviour of compounds **1**–**3** was not fully investigated herein; however, some observations are worth reporting given the similarity of the structures to the widely studied anils. Schiff bases of salicylaldehyde derivatives with aniline derivatives, which exhibit both thermo- and photochromism in the solid state (Cohen & Schmidt, 1962; Cohen *et al.*, 1964; Fujiwara *et al.*, 2004). In anils, a link has been proposed between the dihedral angle (Φ) and the chromic behaviour of some of the Schiff bases, with a suggestion that compounds with $\Phi < 25°$ are expected to be strongly thermochromic, while those with $\Phi > 25°$ are more likely to be photochromic (Hadjoudis & Mavridis, 2004; Robert *et al.*, 2009). Clearly the dihedral angle is not the only factor that has been found to influence chromism in anils, with thermochromic structures tending to be more closely packed than

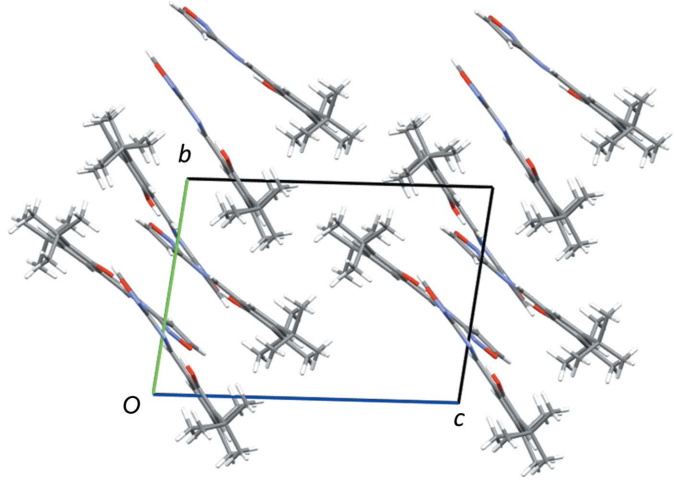


Figure 4
Illustration of the packing in **3**, looking down the *a* axis.

photochromic structures and substituents that weaken the O—H bond or strengthen the accepting ability of the N atom often resulting in more strongly thermochromic complexes (Hadjoudis & Mavridis, 2004; Robert *et al.*, 2009). The Schiff bases of salicylaldehyde derivatives with isoxazole derivatives presented here have not been widely studied in terms of their chromic behaviour and the three compounds presented herein appear to show some differences from the anils. The Φ value was 6.95 (12)° for **1**, 4.42 (14)° for **2** and 6.53 (10)/14.27 (8)° (two molecules) for **3**; however, none of the compounds were observed to be strongly thermochromic by eye when cooled to ∼80 K. In the case of **2** and **3**, this is perhaps not a major surprise as they are yellow at room temperature and, while they did become paler in colour at lower temperatures, the strongly thermochromic anil compounds are typically a red/orange colour at room temperature and change to yellow upon cooling. However, **1**, which is orange at room temperature, remained an orange colour at ∼80 K also. All three compounds did show evidence of photochromism with a colour change, from orange to red for **1** and from yellow to orange for **2** and **3**, upon irradiation with UV light.

4. Conclusion

The structures of three Schiff bases of salicylaldehyde derivatives with isoxazole derivatives, namely, (*E*)-2-{[(isoxazol-3-yl)imino]methyl}phenol, **1**, (*E*)-2-{[(5-methylisoxazol-3-yl)imino]-methyl}phenol, **2**, and (*E*)-2,4-di-*tert*-butyl-6-{[(isoxazol-3-yl)-imino]methyl}phenol, **3**, are reported. The three structures all exist in the enol form and display an intramolecular O—H···N hydrogen bond. All three structures contain intermolecular C—H···N and C—H···O contacts. In the structures of **1** and **2**, π–π-type contacts were identified between the C=N group and the phenol ring. All three compounds had dihedral angles of <25°; however, none of the compounds were observed to be strongly thermochromic and even **1**, which was orange at room temperature, did not show a significant colour change upon cooling. This is in contrast to the anils where orange compounds with a dihedral angle of <25° are normally strongly thermochromic. All three title compounds did show evidence of photochromism upon irradiation with UV light.

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supporting information

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Synthesis and structures of three isoxazole-containing Schiff bases

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Computing details

Data collection: *APEX2* (Bruker, 2012) for (1); *CrysAlis PRO* (Oxford Diffraction, 2010) for (2), (3). Cell refinement: *SAINT* (Bruker, 2012) for (1); *CrysAlis PRO* (Oxford Diffraction, 2010) for (2), (3). Data reduction: *SAINT* (Bruker, 2012) for (1); *CrysAlis PRO* (Oxford Diffraction, 2010) for (2), (3). Program(s) used to solve structure: SHELXT2018 (Sheldrick, 2015a) for (1); *SHELXS97* (Sheldrick, 2008) for (2), (3). For all structures, program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

(*E*)-2-{[(Isoxazol-3-yl)imino]methyl}phenol (1)

Crystal data

$C_{10}H_8N_2O_2$
M_r = 188.18
Orthorhombic, $P2_12_12_1$
a = 4.5999 (5) Å
b = 10.2684 (10) Å
c = 18.711 (2) Å
V = 883.79 (16) Å^3
Z = 4
$F(000)$ = 392
D_x = 1.414 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6240 reflections
θ = 2.3–28.3°
μ = 0.10 mm^{-1}
T = 210 K
Needle, yellow
0.3 × 0.08 × 0.05 mm

Data collection

Bruker SMART APEXII area detector diffractometer
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs
Mirror optics monochromator
Detector resolution: 7.9 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.654, T_{max} = 0.746
10497 measured reflections
2166 independent reflections
1978 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.3°, θ_{min} = 3.0°
h = −5→6
k = −13→13
l = −24→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.08
2166 reflections
131 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.1297P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5619 (3)	0.28237 (12)	0.62081 (8)	0.0437 (3)
H1	0.442 (6)	0.299 (3)	0.6544 (15)	0.076 (9)*
O2	−0.2634 (3)	0.38706 (13)	0.84027 (7)	0.0458 (3)
N1	0.2661 (3)	0.42750 (13)	0.70936 (7)	0.0323 (3)
N2	−0.0828 (4)	0.34323 (14)	0.78487 (8)	0.0416 (4)
C1	0.6881 (4)	0.39393 (18)	0.59858 (9)	0.0348 (4)
C2	0.8989 (4)	0.3867 (2)	0.54531 (9)	0.0440 (4)
H2	0.949799	0.306491	0.525939	0.053*
C3	1.0319 (4)	0.4989 (2)	0.52136 (9)	0.0483 (5)
H3	1.173469	0.493398	0.486002	0.058*
C4	0.9584 (4)	0.6193 (2)	0.54901 (10)	0.0462 (5)
H4	1.048691	0.694275	0.532190	0.055*
C5	0.7498 (4)	0.62710 (18)	0.60178 (10)	0.0389 (4)
H5	0.699931	0.708006	0.620460	0.047*
C6	0.6123 (3)	0.51527 (16)	0.62759 (9)	0.0320 (3)
C7	0.3974 (4)	0.52744 (15)	0.68378 (8)	0.0310 (3)
H7	0.353609	0.609577	0.701746	0.037*
C8	0.0651 (4)	0.44484 (16)	0.76451 (8)	0.0306 (3)
C9	−0.0105 (4)	0.55739 (17)	0.80429 (10)	0.0416 (4)
H9	0.063012	0.641351	0.799683	0.050*
C10	−0.2116 (5)	0.51451 (18)	0.84982 (10)	0.0438 (4)
H10	−0.303966	0.566124	0.883770	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0460 (8)	0.0369 (7)	0.0483 (8)	0.0034 (6)	0.0043 (6)	−0.0077 (5)
O2	0.0545 (8)	0.0400 (7)	0.0428 (7)	−0.0028 (7)	0.0158 (6)	0.0049 (5)
N1	0.0320 (7)	0.0332 (7)	0.0316 (7)	0.0026 (6)	−0.0012 (6)	−0.0007 (5)
N2	0.0484 (9)	0.0338 (7)	0.0426 (8)	−0.0003 (7)	0.0097 (8)	0.0010 (6)
C1	0.0298 (8)	0.0445 (9)	0.0302 (8)	0.0040 (7)	−0.0056 (7)	−0.0028 (7)
C2	0.0346 (9)	0.0635 (11)	0.0338 (9)	0.0065 (9)	−0.0023 (7)	−0.0129 (8)
C3	0.0313 (9)	0.0860 (15)	0.0275 (8)	0.0006 (10)	−0.0013 (7)	−0.0010 (9)
C4	0.0355 (9)	0.0655 (12)	0.0377 (9)	−0.0061 (9)	−0.0018 (8)	0.0123 (9)
C5	0.0340 (9)	0.0441 (9)	0.0385 (9)	−0.0001 (8)	−0.0042 (8)	0.0051 (7)
C6	0.0276 (8)	0.0397 (8)	0.0288 (7)	0.0024 (7)	−0.0049 (6)	0.0009 (6)
C7	0.0309 (8)	0.0320 (7)	0.0302 (8)	0.0050 (7)	−0.0030 (6)	−0.0013 (6)
C8	0.0324 (8)	0.0306 (7)	0.0290 (8)	0.0020 (6)	−0.0028 (6)	0.0022 (6)
C9	0.0531 (11)	0.0320 (8)	0.0395 (9)	−0.0041 (8)	0.0097 (9)	−0.0043 (7)

C10	0.0567 (12)	0.0396 (9)	0.0352 (8)	−0.0005 (8)	0.0117 (8)	−0.0020 (7)

Geometric parameters (Å, º)

O1—H1	0.85 (3)	C3—C4	1.382 (3)
O1—C1	1.350 (2)	C4—H4	0.9300
O2—N2	1.403 (2)	C4—C5	1.379 (3)
O2—C10	1.342 (2)	C5—H5	0.9300
N1—C7	1.283 (2)	C5—C6	1.397 (2)
N1—C8	1.397 (2)	C6—C7	1.449 (2)
N2—C8	1.302 (2)	C7—H7	0.9300
C1—C2	1.392 (2)	C8—C9	1.418 (2)
C1—C6	1.403 (2)	C9—H9	0.9300
C2—H2	0.9300	C9—C10	1.333 (3)
C2—C3	1.379 (3)	C10—H10	0.9300
C3—H3	0.9300		
C1—O1—H1	109.5 (19)	C4—C5—C6	120.96 (18)
C10—O2—N2	107.82 (13)	C6—C5—H5	119.5
C7—N1—C8	119.02 (14)	C1—C6—C7	121.80 (15)
C8—N2—O2	105.57 (13)	C5—C6—C1	118.93 (16)
O1—C1—C2	118.36 (17)	C5—C6—C7	119.27 (15)
O1—C1—C6	121.83 (15)	N1—C7—C6	121.53 (15)
C2—C1—C6	119.82 (17)	N1—C7—H7	119.2
C1—C2—H2	120.1	C6—C7—H7	119.2
C3—C2—C1	119.81 (18)	N1—C8—C9	130.84 (16)
C3—C2—H2	120.1	N2—C8—N1	117.36 (14)
C2—C3—H3	119.4	N2—C8—C9	111.80 (15)
C2—C3—C4	121.11 (17)	C8—C9—H9	128.2
C4—C3—H3	119.4	C10—C9—C8	103.68 (16)
C3—C4—H4	120.3	C10—C9—H9	128.2
C5—C4—C3	119.37 (19)	O2—C10—H10	124.4
C5—C4—H4	120.3	C9—C10—O2	111.12 (16)
C4—C5—H5	119.5	C9—C10—H10	124.4
O1—C1—C2—C3	179.96 (16)	C2—C3—C4—C5	0.4 (3)
O1—C1—C6—C5	−179.52 (15)	C3—C4—C5—C6	0.0 (3)
O1—C1—C6—C7	0.9 (2)	C4—C5—C6—C1	−0.5 (2)
O2—N2—C8—N1	179.55 (13)	C4—C5—C6—C7	179.11 (15)
O2—N2—C8—C9	−0.3 (2)	C5—C6—C7—N1	−179.78 (15)
N1—C8—C9—C10	−179.35 (18)	C6—C1—C2—C3	0.0 (2)
N2—O2—C10—C9	0.3 (2)	C7—N1—C8—N2	174.09 (16)
N2—C8—C9—C10	0.5 (2)	C7—N1—C8—C9	−6.1 (3)
C1—C2—C3—C4	−0.4 (3)	C8—N1—C7—C6	178.73 (14)
C1—C6—C7—N1	−0.2 (2)	C8—C9—C10—O2	−0.4 (2)
C2—C1—C6—C5	0.4 (2)	C10—O2—N2—C8	0.01 (19)
C2—C1—C6—C7	−179.10 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.85 (3)	1.86 (3)	2.6110 (19)	146 (3)
C7—H7···N2^{i}	0.93	2.71	3.599 (2)	159
C9—H9···O1^{ii}	0.93	2.70	3.400 (2)	133
C9—H9···N2^{i}	0.93	2.61	3.403 (2)	144
C10—H10···O1^{i}	0.93	2.52	3.235 (2)	134

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+3/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2.

(*E*)-2-{[(5-Methylisoxazol-3-yl)imino]methyl}phenol (2)

Crystal data

$C_{11}H_{10}N_2O_2$
M_r = 202.21
Orthorhombic, $Pna2_1$
a = 20.5584 (7) Å
b = 10.0468 (4) Å
c = 4.6417 (2) Å
V = 958.73 (7) Å^3
Z = 4
F(000) = 424
D_x = 1.401 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1956 reflections
θ = 2.8–30.4°
μ = 0.10 mm^{-1}
T = 120 K
Plate, yellow
0.49 × 0.24 × 0.09 mm

Data collection

Oxford Diffraction Xcalibur (Sapphire3, Gemini ultra) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1511 pixels mm^{-1}
ω scans
Absorption correction: analytical (CrysAlis PRO; Oxford Diffraction, 2010)
T_{min} = 0.969, T_{max} = 0.991
6756 measured reflections
2021 independent reflections
1819 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.1°, θ_{min} = 2.8°
h = −26→24
k = −7→12
l = −5→5

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.081
S = 1.05
2021 reflections
141 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 0.0649P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.81572 (8)	0.82495 (18)	0.5857 (4)	0.0178 (4)
O2	0.69696 (7)	0.88359 (16)	1.1087 (4)	0.0252 (4)
C5	0.89944 (10)	0.6016 (2)	0.0907 (6)	0.0230 (5)
H5	0.876479	0.522402	0.137253	0.028*
C8	0.76594 (9)	0.8126 (2)	0.7920 (5)	0.0174 (5)
C7	0.83312 (10)	0.7187 (2)	0.4495 (5)	0.0171 (5)
H7	0.812090	0.636888	0.492966	0.021*
C6	0.88369 (10)	0.7205 (2)	0.2328 (4)	0.0185 (5)
C9	0.73150 (10)	0.7000 (2)	0.8961 (5)	0.0190 (5)
H9	0.737117	0.609553	0.841572	0.023*
N2	0.74662 (9)	0.9233 (2)	0.9160 (4)	0.0254 (5)
C10	0.68911 (10)	0.7496 (2)	1.0894 (5)	0.0187 (5)
C2	0.96625 (11)	0.8330 (3)	−0.0493 (5)	0.0280 (6)
H2	0.989209	0.911688	−0.099409	0.034*
C1	0.91752 (10)	0.8371 (2)	0.1593 (5)	0.0212 (5)
C4	0.94762 (11)	0.5978 (3)	−0.1150 (5)	0.0282 (6)
H4	0.957931	0.516631	−0.209719	0.034*
C3	0.98101 (10)	0.7139 (3)	−0.1827 (5)	0.0294 (6)
H3	1.014582	0.711294	−0.323261	0.035*
C11	0.63763 (10)	0.6919 (2)	1.2750 (5)	0.0246 (5)
H11A	0.649494	0.600596	1.327622	0.037*
H11B	0.596262	0.691209	1.170093	0.037*
H11C	0.633106	0.745691	1.449976	0.037*
O1	0.90403 (8)	0.95508 (17)	0.2871 (4)	0.0271 (4)
H1	0.8719 (16)	0.943 (3)	0.431 (7)	0.062 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0184 (9)	0.0183 (10)	0.0167 (9)	−0.0007 (7)	−0.0013 (7)	0.0014 (8)
O2	0.0273 (8)	0.0198 (8)	0.0286 (9)	0.0038 (7)	0.0080 (7)	−0.0025 (7)
C5	0.0234 (11)	0.0234 (13)	0.0221 (12)	0.0041 (9)	−0.0012 (10)	0.0035 (10)
C8	0.0166 (10)	0.0172 (11)	0.0184 (11)	0.0019 (8)	−0.0032 (9)	−0.0008 (9)
C7	0.0177 (10)	0.0162 (11)	0.0175 (11)	−0.0015 (9)	−0.0037 (8)	−0.0005 (9)
C6	0.0166 (10)	0.0228 (12)	0.0162 (12)	−0.0011 (9)	−0.0035 (8)	0.0019 (9)
C9	0.0212 (11)	0.0143 (11)	0.0216 (12)	−0.0009 (9)	−0.0004 (9)	−0.0041 (9)
N2	0.0278 (10)	0.0191 (10)	0.0293 (12)	0.0008 (8)	0.0081 (9)	−0.0017 (9)
C10	0.0178 (10)	0.0185 (11)	0.0197 (12)	0.0004 (9)	−0.0057 (8)	−0.0004 (10)
C2	0.0181 (11)	0.0425 (17)	0.0232 (13)	−0.0075 (10)	−0.0040 (9)	0.0097 (12)
C1	0.0191 (10)	0.0252 (13)	0.0194 (12)	−0.0019 (9)	−0.0057 (9)	0.0019 (10)
C4	0.0264 (12)	0.0376 (16)	0.0206 (12)	0.0107 (10)	−0.0008 (10)	0.0004 (11)
C3	0.0168 (11)	0.0502 (18)	0.0210 (12)	0.0031 (11)	0.0009 (9)	0.0050 (12)
C11	0.0208 (11)	0.0287 (14)	0.0243 (12)	−0.0006 (10)	0.0020 (10)	−0.0030 (11)
O1	0.0297 (9)	0.0228 (9)	0.0287 (9)	−0.0084 (7)	0.0002 (8)	0.0016 (8)

Geometric parameters (Å, º)

N1—C8	1.407 (3)	C9—C10	1.347 (3)
N1—C7	1.291 (3)	C10—C11	1.483 (3)
O2—N2	1.415 (2)	C2—H2	0.9500
O2—C10	1.358 (3)	C2—C1	1.394 (3)
C5—H5	0.9500	C2—C3	1.381 (4)
C5—C6	1.402 (3)	C1—O1	1.354 (3)
C5—C4	1.377 (3)	C4—H4	0.9500
C8—C9	1.420 (3)	C4—C3	1.389 (4)
C8—N2	1.314 (3)	C3—H3	0.9500
C7—H7	0.9500	C11—H11A	0.9800
C7—C6	1.447 (3)	C11—H11B	0.9800
C6—C1	1.405 (3)	C11—H11C	0.9800
C9—H9	0.9500	O1—H1	0.95 (3)
C7—N1—C8	117.49 (18)	C9—C10—C11	134.7 (2)
C10—O2—N2	108.88 (17)	C1—C2—H2	120.1
C6—C5—H5	119.5	C3—C2—H2	120.1
C4—C5—H5	119.5	C3—C2—C1	119.7 (2)
C4—C5—C6	121.1 (2)	C2—C1—C6	120.0 (2)
N1—C8—C9	131.7 (2)	O1—C1—C6	121.5 (2)
N2—C8—N1	116.30 (19)	O1—C1—C2	118.6 (2)
N2—C8—C9	112.03 (19)	C5—C4—H4	120.4
N1—C7—H7	119.0	C5—C4—C3	119.3 (2)
N1—C7—C6	121.9 (2)	C3—C4—H4	120.4
C6—C7—H7	119.0	C2—C3—C4	121.2 (2)
C5—C6—C7	118.8 (2)	C2—C3—H3	119.4
C5—C6—C1	118.8 (2)	C4—C3—H3	119.4
C1—C6—C7	122.3 (2)	C10—C11—H11A	109.5
C8—C9—H9	127.6	C10—C11—H11B	109.5
C10—C9—C8	104.72 (19)	C10—C11—H11C	109.5
C10—C9—H9	127.6	H11A—C11—H11B	109.5
C8—N2—O2	104.84 (18)	H11A—C11—H11C	109.5
O2—C10—C11	115.73 (19)	H11B—C11—H11C	109.5
C9—C10—O2	109.53 (19)	C1—O1—H1	110 (2)
N1—C8—C9—C10	−179.3 (2)	C7—C6—C1—O1	−0.5 (3)
N1—C8—N2—O2	179.62 (17)	C6—C5—C4—C3	−0.1 (3)
N1—C7—C6—C5	−179.1 (2)	C9—C8—N2—O2	−0.5 (2)
N1—C7—C6—C1	0.8 (3)	N2—O2—C10—C9	0.6 (2)
C5—C6—C1—C2	−0.7 (3)	N2—O2—C10—C11	−178.90 (17)
C5—C6—C1—O1	179.4 (2)	N2—C8—C9—C10	0.9 (2)
C5—C4—C3—C2	−0.6 (3)	C10—O2—N2—C8	0.0 (2)
C8—N1—C7—C6	179.86 (18)	C1—C2—C3—C4	0.7 (4)
C8—C9—C10—O2	−0.8 (2)	C4—C5—C6—C7	−179.34 (19)
C8—C9—C10—C11	178.5 (2)	C4—C5—C6—C1	0.8 (3)
C7—N1—C8—C9	3.8 (3)	C3—C2—C1—C6	0.0 (3)

C7—N1—C8—N2	−176.33 (19)	C3—C2—C1—O1	179.9 (2)
C7—C6—C1—C2	179.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O2^{i}	0.95	2.61	3.502 (3)	157
C7—H7···N2^{i}	0.95	2.49	3.394 (3)	159
C9—H9···N2^{i}	0.95	2.74	3.591 (3)	149
C2—H2···O1^{ii}	0.95	2.62	3.496 (3)	153
O1—H1···N1	0.95 (3)	1.80 (3)	2.632 (2)	145 (3)

Symmetry codes: (i) −*x*+3/2, *y*−1/2, *z*−1/2; (ii) −*x*+2, −*y*+2, *z*−1/2.

(*E*)-2,4-Di-*tert*-butyl-6-{[(isoxazol-3-yl)imino]methyl}phenol (3)

Crystal data

$C_{18}H_{24}N_2O_2$
M_r = 300.39
Triclinic, $P\overline{1}$
a = 10.8955 (5) Å
b = 10.9571 (4) Å
c = 14.8329 (6) Å
α = 82.335 (3)°
β = 88.326 (4)°
γ = 75.178 (3)°
V = 1696.56 (12) Å^3
Z = 4
F(000) = 648
D_x = 1.176 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3727 reflections
θ = 2.8–30.6°
μ = 0.08 mm^{-1}
T = 120 K
Block, yellow
0.6 × 0.31 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur (Sapphire3, Gemini ultra) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1511 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Oxford Diffraction, 201)
T_{min} = 0.833, T_{max} = 1.000
14901 measured reflections
6942 independent reflections
5078 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.4°, θ_{min} = 2.8°
h = −13→13
k = −13→13
l = −17→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.119
S = 1.02
6942 reflections
447 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.2797P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C24	0.62533 (14)	0.44081 (15)	0.23482 (11)	0.0194 (3)	
C20	0.77675 (14)	0.32155 (14)	0.35354 (11)	0.0190 (3)	
C19	0.75097 (14)	0.39906 (15)	0.26936 (11)	0.0199 (4)	
C23	0.52635 (14)	0.40545 (15)	0.28547 (11)	0.0205 (4)	
H23	0.442462	0.433070	0.261467	0.025*	
C21	0.67363 (14)	0.29147 (15)	0.40078 (11)	0.0204 (4)	
H21	0.689707	0.240305	0.458239	0.024*	
C22	0.54747 (14)	0.33172 (15)	0.36914 (11)	0.0197 (4)	
C25	0.59372 (14)	0.52312 (14)	0.14975 (11)	0.0200 (4)	
H25	0.507954	0.547471	0.129459	0.024*	
C33	0.43854 (15)	0.29616 (16)	0.42524 (12)	0.0252 (4)	
C26	0.63774 (14)	0.64763 (15)	0.02015 (11)	0.0194 (3)	
C29	0.91235 (14)	0.27455 (15)	0.39314 (12)	0.0215 (4)	
C32	0.91766 (16)	0.18777 (18)	0.48356 (13)	0.0317 (4)	
H32A	0.867081	0.235952	0.528972	0.048*	
H32B	1.005954	0.156148	0.504530	0.048*	
H32C	0.883353	0.115653	0.475082	0.048*	
C31	0.96017 (17)	0.38940 (17)	0.41060 (14)	0.0324 (4)	
H31A	0.961864	0.444585	0.353107	0.049*	
H31B	1.045981	0.359682	0.436576	0.049*	
H31C	0.903225	0.437328	0.453275	0.049*	
C30	1.00116 (15)	0.19654 (16)	0.32725 (13)	0.0276 (4)	
H30A	0.972608	0.120478	0.320148	0.041*	
H30B	1.087901	0.170943	0.351673	0.041*	
H30C	0.999547	0.248499	0.267943	0.041*	
C2	−0.07843 (14)	−0.06923 (15)	0.20567 (11)	0.0192 (3)	
C3	0.04914 (14)	−0.12430 (15)	0.22980 (11)	0.0199 (4)	
H3A	0.068023	−0.198817	0.273037	0.024*	
C1	−0.10414 (14)	0.04067 (15)	0.14176 (11)	0.0204 (4)	
C6	−0.00444 (14)	0.09006 (15)	0.10284 (11)	0.0207 (4)	
C4	0.15019 (14)	−0.07657 (15)	0.19430 (11)	0.0189 (3)	
C5	0.12126 (14)	0.03076 (15)	0.13060 (11)	0.0213 (4)	
H5	0.187942	0.065334	0.104933	0.026*	
C11	−0.18571 (15)	−0.12427 (16)	0.24995 (12)	0.0228 (4)	
C15	0.28885 (14)	−0.13476 (15)	0.22475 (12)	0.0217 (4)	
C7	−0.02506 (15)	0.20119 (15)	0.03592 (11)	0.0224 (4)	
H7	0.046380	0.229436	0.012331	0.027*	
C8	−0.14592 (14)	0.36909 (15)	−0.06000 (11)	0.0207 (4)	
C16	0.37378 (15)	−0.15129 (16)	0.14078 (12)	0.0269 (4)	

H16A	0.369366	−0.067732	0.105930	0.040*	
H16B	0.344641	−0.204890	0.102631	0.040*	
H16C	0.461663	−0.191826	0.160058	0.040*	
C18	0.30689 (15)	−0.26528 (16)	0.28202 (12)	0.0268 (4)	
H18A	0.397268	−0.301710	0.295934	0.040*	
H18B	0.275644	−0.322012	0.247915	0.040*	
H18C	0.259299	−0.255599	0.338826	0.040*	
C14	−0.13416 (16)	−0.24950 (16)	0.31207 (13)	0.0306 (4)	
H14A	−0.079092	−0.311489	0.276944	0.046*	
H14B	−0.205110	−0.282875	0.337046	0.046*	
H14C	−0.085291	−0.234256	0.361952	0.046*	
C17	0.32965 (16)	−0.04451 (17)	0.28165 (13)	0.0307 (4)	
H17A	0.318299	0.039444	0.245718	0.046*	
H17B	0.419187	−0.078850	0.298978	0.046*	
H17C	0.277510	−0.036524	0.336559	0.046*	
C13	−0.26844 (16)	−0.02779 (17)	0.30742 (13)	0.0325 (4)	
H13A	−0.217373	−0.015532	0.357149	0.049*	
H13B	−0.339930	−0.060032	0.332690	0.049*	
H13C	−0.300756	0.053696	0.269041	0.049*	
C12	−0.26677 (17)	−0.15318 (18)	0.17645 (13)	0.0338 (4)	
H12A	−0.332835	−0.190838	0.205501	0.051*	
H12B	−0.212677	−0.213102	0.139369	0.051*	
H12C	−0.306501	−0.074053	0.137610	0.051*	
O1	−0.22667 (10)	0.10067 (12)	0.11699 (9)	0.0277 (3)	
H1	−0.221 (2)	0.169 (2)	0.0752 (16)	0.058 (7)*	
N1	−0.13695 (12)	0.26419 (12)	0.00656 (9)	0.0221 (3)	
N2	−0.25886 (12)	0.42300 (12)	−0.09629 (10)	0.0227 (3)	
N3	0.67760 (12)	0.56493 (12)	0.10016 (9)	0.0210 (3)	
O3	0.84641 (10)	0.43463 (12)	0.22084 (9)	0.0266 (3)	
H3	0.811 (2)	0.481 (2)	0.1680 (18)	0.072 (8)*	
O4	0.65569 (11)	0.78296 (11)	−0.09613 (8)	0.0283 (3)	
O2	−0.24050 (10)	0.52280 (10)	−0.16117 (8)	0.0253 (3)	
C9	−0.05245 (15)	0.43050 (16)	−0.09767 (12)	0.0266 (4)	
H9	0.035303	0.410046	−0.082817	0.032*	
N4	0.71975 (13)	0.70662 (13)	−0.01941 (10)	0.0248 (3)	
C27	0.51994 (15)	0.68092 (15)	−0.02802 (11)	0.0224 (4)	
H27	0.446363	0.650925	−0.013571	0.027*	
C10	−0.11690 (15)	0.52365 (16)	−0.15882 (12)	0.0275 (4)	
H10	−0.080578	0.582833	−0.195853	0.033*	
C28	0.53779 (15)	0.76434 (16)	−0.09833 (12)	0.0260 (4)	
H28	0.475889	0.805189	−0.143839	0.031*	
C34A	0.3752 (3)	0.2222 (3)	0.3685 (2)	0.0348 (8)	0.595
H34A	0.436497	0.143182	0.357110	0.052*	0.595
H34B	0.346150	0.274214	0.310443	0.052*	0.595
H34C	0.302420	0.201712	0.401744	0.052*	0.595
C35A	0.3371 (3)	0.4226 (3)	0.4403 (3)	0.0402 (9)	0.595
H35A	0.265856	0.401680	0.475304	0.060*	0.595
H35B	0.306157	0.470294	0.381160	0.060*	0.595

H35C	0.376041	0.474470	0.473686	0.060*	0.595
C36A	0.4795 (3)	0.2210 (4)	0.5141 (3)	0.0481 (10)	0.595
H36A	0.405423	0.203601	0.547222	0.072*	0.595
H36B	0.520614	0.269126	0.549358	0.072*	0.595
H36C	0.539682	0.140448	0.504702	0.072*	0.595
C34B	0.4739 (5)	0.1446 (5)	0.4570 (4)	0.0401 (12)	0.405
H34D	0.555023	0.118903	0.489858	0.060*	0.405
H34E	0.480813	0.099833	0.403387	0.060*	0.405
H34F	0.407183	0.123146	0.496987	0.060*	0.405
C36B	0.3150 (4)	0.3263 (6)	0.3762 (4)	0.0475 (15)	0.405
H36D	0.253067	0.293945	0.415242	0.071*	0.405
H36E	0.326514	0.286031	0.320331	0.071*	0.405
H36F	0.283947	0.418755	0.360766	0.071*	0.405
C35B	0.4259 (5)	0.3583 (6)	0.5127 (4)	0.0488 (14)	0.405
H35D	0.358193	0.334260	0.550231	0.073*	0.405
H35E	0.405194	0.451094	0.497311	0.073*	0.405
H35F	0.506299	0.329347	0.546512	0.073*	0.405

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C24	0.0181 (8)	0.0219 (8)	0.0182 (9)	−0.0050 (6)	−0.0009 (6)	−0.0032 (7)
C20	0.0172 (8)	0.0203 (8)	0.0198 (9)	−0.0039 (6)	−0.0018 (6)	−0.0055 (7)
C19	0.0173 (8)	0.0229 (8)	0.0206 (9)	−0.0068 (6)	0.0028 (7)	−0.0042 (7)
C23	0.0132 (7)	0.0253 (8)	0.0226 (9)	−0.0042 (6)	−0.0007 (6)	−0.0030 (7)
C21	0.0209 (8)	0.0217 (8)	0.0174 (9)	−0.0036 (7)	−0.0008 (7)	−0.0019 (7)
C22	0.0180 (8)	0.0211 (8)	0.0208 (9)	−0.0063 (6)	0.0007 (7)	−0.0029 (7)
C25	0.0172 (8)	0.0220 (8)	0.0206 (9)	−0.0038 (6)	−0.0005 (7)	−0.0040 (7)
C33	0.0206 (8)	0.0326 (9)	0.0232 (10)	−0.0095 (7)	0.0029 (7)	−0.0018 (8)
C26	0.0210 (8)	0.0210 (8)	0.0167 (9)	−0.0060 (7)	0.0019 (7)	−0.0038 (7)
C29	0.0163 (8)	0.0257 (9)	0.0224 (10)	−0.0041 (7)	−0.0025 (7)	−0.0050 (7)
C32	0.0209 (9)	0.0405 (11)	0.0295 (11)	−0.0015 (8)	−0.0072 (8)	0.0002 (8)
C31	0.0258 (9)	0.0326 (10)	0.0405 (12)	−0.0063 (8)	−0.0091 (8)	−0.0114 (9)
C30	0.0191 (8)	0.0304 (9)	0.0320 (11)	−0.0023 (7)	0.0006 (7)	−0.0077 (8)
C2	0.0183 (8)	0.0242 (8)	0.0171 (9)	−0.0079 (7)	0.0012 (6)	−0.0055 (7)
C3	0.0216 (8)	0.0208 (8)	0.0170 (9)	−0.0045 (7)	−0.0015 (7)	−0.0022 (7)
C1	0.0143 (7)	0.0248 (9)	0.0220 (9)	−0.0039 (6)	−0.0018 (7)	−0.0043 (7)
C6	0.0180 (8)	0.0243 (9)	0.0200 (9)	−0.0061 (7)	−0.0003 (7)	−0.0021 (7)
C4	0.0158 (7)	0.0223 (8)	0.0190 (9)	−0.0042 (6)	0.0005 (6)	−0.0060 (7)
C5	0.0158 (8)	0.0271 (9)	0.0213 (9)	−0.0065 (7)	0.0021 (7)	−0.0027 (7)
C11	0.0196 (8)	0.0272 (9)	0.0239 (10)	−0.0106 (7)	−0.0003 (7)	−0.0017 (7)
C15	0.0157 (8)	0.0255 (9)	0.0235 (10)	−0.0033 (7)	−0.0028 (7)	−0.0051 (7)
C7	0.0167 (8)	0.0287 (9)	0.0221 (10)	−0.0070 (7)	0.0008 (7)	−0.0023 (7)
C8	0.0177 (8)	0.0225 (8)	0.0213 (9)	−0.0038 (7)	0.0007 (7)	−0.0039 (7)
C16	0.0179 (8)	0.0303 (9)	0.0302 (11)	−0.0022 (7)	0.0006 (7)	−0.0036 (8)
C18	0.0202 (8)	0.0312 (9)	0.0257 (10)	−0.0002 (7)	−0.0035 (7)	−0.0034 (8)
C14	0.0279 (9)	0.0333 (10)	0.0329 (11)	−0.0152 (8)	0.0009 (8)	0.0023 (8)
C17	0.0244 (9)	0.0318 (10)	0.0350 (12)	−0.0025 (8)	−0.0093 (8)	−0.0089 (8)

C13	0.0270 (9)	0.0379 (10)	0.0331 (12)	−0.0104 (8)	0.0108 (8)	−0.0040 (9)
C12	0.0280 (9)	0.0409 (11)	0.0380 (12)	−0.0185 (8)	−0.0043 (8)	−0.0051 (9)
O1	0.0145 (6)	0.0327 (7)	0.0325 (8)	−0.0054 (5)	−0.0020 (5)	0.0075 (6)
N1	0.0193 (7)	0.0243 (7)	0.0218 (8)	−0.0054 (6)	−0.0008 (6)	0.0005 (6)
N2	0.0214 (7)	0.0229 (7)	0.0233 (8)	−0.0072 (6)	−0.0024 (6)	0.0018 (6)
N3	0.0212 (7)	0.0244 (7)	0.0177 (8)	−0.0069 (6)	−0.0007 (6)	−0.0017 (6)
O3	0.0166 (6)	0.0384 (7)	0.0246 (7)	−0.0099 (5)	0.0008 (5)	0.0027 (6)
O4	0.0296 (6)	0.0304 (7)	0.0239 (7)	−0.0097 (5)	−0.0018 (5)	0.0041 (5)
O2	0.0241 (6)	0.0264 (6)	0.0240 (7)	−0.0068 (5)	−0.0030 (5)	0.0032 (5)
C9	0.0170 (8)	0.0305 (9)	0.0305 (11)	−0.0061 (7)	0.0008 (7)	0.0019 (8)
N4	0.0265 (7)	0.0281 (8)	0.0194 (8)	−0.0086 (6)	−0.0026 (6)	0.0016 (6)
C27	0.0198 (8)	0.0256 (9)	0.0217 (10)	−0.0052 (7)	0.0002 (7)	−0.0035 (7)
C10	0.0212 (9)	0.0316 (10)	0.0292 (11)	−0.0093 (7)	0.0030 (7)	0.0018 (8)
C28	0.0213 (8)	0.0282 (9)	0.0267 (10)	−0.0030 (7)	−0.0026 (7)	−0.0026 (8)
C34A	0.0294 (16)	0.0399 (19)	0.041 (2)	−0.0199 (15)	0.0049 (15)	−0.0064 (16)
C35A	0.0336 (17)	0.0361 (18)	0.053 (2)	−0.0126 (15)	0.0223 (17)	−0.0109 (17)
C36A	0.0302 (18)	0.076 (3)	0.037 (2)	−0.0265 (19)	−0.0025 (15)	0.024 (2)
C34B	0.036 (3)	0.038 (3)	0.048 (3)	−0.019 (2)	0.011 (2)	0.005 (2)
C36B	0.022 (2)	0.074 (4)	0.045 (3)	−0.021 (3)	0.001 (2)	0.014 (3)
C35B	0.045 (3)	0.062 (4)	0.050 (4)	−0.030 (3)	0.026 (3)	−0.020 (3)

Geometric parameters (Å, º)

C24—C19	1.416 (2)	C7—H7	0.9500
C24—C23	1.402 (2)	C7—N1	1.2933 (19)
C24—C25	1.445 (2)	C8—N1	1.397 (2)
C20—C19	1.405 (2)	C8—N2	1.3172 (19)
C20—C21	1.394 (2)	C8—C9	1.421 (2)
C20—C29	1.541 (2)	C16—H16A	0.9800
C19—O3	1.3564 (19)	C16—H16B	0.9800
C23—H23	0.9500	C16—H16C	0.9800
C23—C22	1.378 (2)	C18—H18A	0.9800
C21—H21	0.9500	C18—H18B	0.9800
C21—C22	1.406 (2)	C18—H18C	0.9800
C22—C33	1.531 (2)	C14—H14A	0.9800
C25—H25	0.9500	C14—H14B	0.9800
C25—N3	1.293 (2)	C14—H14C	0.9800
C33—C34A	1.531 (4)	C17—H17A	0.9800
C33—C35A	1.573 (3)	C17—H17B	0.9800
C33—C36A	1.473 (4)	C17—H17C	0.9800
C33—C34B	1.613 (5)	C13—H13A	0.9800
C33—C36B	1.489 (5)	C13—H13B	0.9800
C33—C35B	1.530 (6)	C13—H13C	0.9800
C26—N3	1.400 (2)	C12—H12A	0.9800
C26—N4	1.313 (2)	C12—H12B	0.9800
C26—C27	1.427 (2)	C12—H12C	0.9800
C29—C32	1.531 (2)	O1—H1	0.92 (2)
C29—C31	1.534 (2)	N2—O2	1.4078 (17)

C29—C30	1.538 (2)	O3—H3	0.91 (3)
C32—H32A	0.9800	O4—N4	1.4056 (17)
C32—H32B	0.9800	O4—C28	1.353 (2)
C32—H32C	0.9800	O2—C10	1.3507 (19)
C31—H31A	0.9800	C9—H9	0.9500
C31—H31B	0.9800	C9—C10	1.335 (2)
C31—H31C	0.9800	C27—H27	0.9500
C30—H30A	0.9800	C27—C28	1.336 (2)
C30—H30B	0.9800	C10—H10	0.9500
C30—H30C	0.9800	C28—H28	0.9500
C2—C3	1.402 (2)	C34A—H34A	0.9800
C2—C1	1.402 (2)	C34A—H34B	0.9800
C2—C11	1.540 (2)	C34A—H34C	0.9800
C3—H3A	0.9500	C35A—H35A	0.9800
C3—C4	1.396 (2)	C35A—H35B	0.9800
C1—C6	1.410 (2)	C35A—H35C	0.9800
C1—O1	1.3655 (18)	C36A—H36A	0.9800
C6—C5	1.405 (2)	C36A—H36B	0.9800
C6—C7	1.439 (2)	C36A—H36C	0.9800
C4—C5	1.381 (2)	C34B—H34D	0.9800
C4—C15	1.537 (2)	C34B—H34E	0.9800
C5—H5	0.9500	C34B—H34F	0.9800
C11—C14	1.532 (2)	C36B—H36D	0.9800
C11—C13	1.538 (2)	C36B—H36E	0.9800
C11—C12	1.536 (2)	C36B—H36F	0.9800
C15—C16	1.532 (2)	C35B—H35D	0.9800
C15—C18	1.532 (2)	C35B—H35E	0.9800
C15—C17	1.535 (2)	C35B—H35F	0.9800

C19—C24—C25	122.25 (15)	N2—C8—N1	116.85 (14)
C23—C24—C19	119.65 (15)	N2—C8—C9	112.00 (14)
C23—C24—C25	118.06 (14)	C15—C16—H16A	109.5
C19—C20—C29	121.58 (14)	C15—C16—H16B	109.5
C21—C20—C19	117.03 (14)	C15—C16—H16C	109.5
C21—C20—C29	121.37 (14)	H16A—C16—H16B	109.5
C20—C19—C24	120.26 (14)	H16A—C16—H16C	109.5
O3—C19—C24	119.60 (15)	H16B—C16—H16C	109.5
O3—C19—C20	120.14 (14)	C15—C18—H18A	109.5
C24—C23—H23	119.1	C15—C18—H18B	109.5
C22—C23—C24	121.79 (14)	C15—C18—H18C	109.5
C22—C23—H23	119.1	H18A—C18—H18B	109.5
C20—C21—H21	117.8	H18A—C18—H18C	109.5
C20—C21—C22	124.44 (15)	H18B—C18—H18C	109.5
C22—C21—H21	117.8	C11—C14—H14A	109.5
C23—C22—C21	116.82 (15)	C11—C14—H14B	109.5
C23—C22—C33	121.57 (14)	C11—C14—H14C	109.5
C21—C22—C33	121.61 (15)	H14A—C14—H14B	109.5
C24—C25—H25	118.7	H14A—C14—H14C	109.5

N3—C25—C24	122.57 (14)	H14B—C14—H14C	109.5
N3—C25—H25	118.7	C15—C17—H17A	109.5
C22—C33—C35A	108.26 (17)	C15—C17—H17B	109.5
C22—C33—C34B	109.7 (2)	C15—C17—H17C	109.5
C34A—C33—C22	107.78 (17)	H17A—C17—H17B	109.5
C34A—C33—C35A	107.4 (2)	H17A—C17—H17C	109.5
C36A—C33—C22	113.46 (17)	H17B—C17—H17C	109.5
C36A—C33—C34A	110.3 (2)	C11—C13—H13A	109.5
C36A—C33—C35A	109.4 (2)	C11—C13—H13B	109.5
C36B—C33—C22	115.0 (2)	C11—C13—H13C	109.5
C36B—C33—C34B	105.7 (3)	H13A—C13—H13B	109.5
C36B—C33—C35B	111.5 (3)	H13A—C13—H13C	109.5
C35B—C33—C22	108.5 (2)	H13B—C13—H13C	109.5
C35B—C33—C34B	106.0 (3)	C11—C12—H12A	109.5
N3—C26—C27	130.88 (15)	C11—C12—H12B	109.5
N4—C26—N3	116.92 (14)	C11—C12—H12C	109.5
N4—C26—C27	112.19 (14)	H12A—C12—H12B	109.5
C32—C29—C20	112.13 (13)	H12A—C12—H12C	109.5
C32—C29—C31	107.71 (14)	H12B—C12—H12C	109.5
C32—C29—C30	106.86 (13)	C1—O1—H1	105.1 (14)
C31—C29—C20	109.32 (13)	C7—N1—C8	117.91 (14)
C31—C29—C30	110.55 (14)	C8—N2—O2	105.03 (12)
C30—C29—C20	110.24 (13)	C25—N3—C26	118.67 (13)
C29—C32—H32A	109.5	C19—O3—H3	106.6 (16)
C29—C32—H32B	109.5	C28—O4—N4	108.24 (12)
C29—C32—H32C	109.5	C10—O2—N2	108.06 (12)
H32A—C32—H32B	109.5	C8—C9—H9	128.1
H32A—C32—H32C	109.5	C10—C9—C8	103.75 (14)
H32B—C32—H32C	109.5	C10—C9—H9	128.1
C29—C31—H31A	109.5	C26—N4—O4	105.04 (12)
C29—C31—H31B	109.5	C26—C27—H27	128.3
C29—C31—H31C	109.5	C28—C27—C26	103.47 (15)
H31A—C31—H31B	109.5	C28—C27—H27	128.3
H31A—C31—H31C	109.5	O2—C10—H10	124.4
H31B—C31—H31C	109.5	C9—C10—O2	111.16 (15)
C29—C30—H30A	109.5	C9—C10—H10	124.4
C29—C30—H30B	109.5	O4—C28—H28	124.5
C29—C30—H30C	109.5	C27—C28—O4	111.05 (15)
H30A—C30—H30B	109.5	C27—C28—H28	124.5
H30A—C30—H30C	109.5	C33—C34A—H34A	109.5
H30B—C30—H30C	109.5	C33—C34A—H34B	109.5
C3—C2—C11	121.46 (14)	C33—C34A—H34C	109.5
C1—C2—C3	116.96 (14)	H34A—C34A—H34B	109.5
C1—C2—C11	121.55 (13)	H34A—C34A—H34C	109.5
C2—C3—H3A	117.9	H34B—C34A—H34C	109.5
C4—C3—C2	124.17 (15)	C33—C35A—H35A	109.5
C4—C3—H3A	117.9	C33—C35A—H35B	109.5
C2—C1—C6	120.53 (13)	C33—C35A—H35C	109.5

O1—C1—C2	119.87 (14)	H35A—C35A—H35B	109.5
O1—C1—C6	119.60 (14)	H35A—C35A—H35C	109.5
C1—C6—C7	122.93 (14)	H35B—C35A—H35C	109.5
C5—C6—C1	119.57 (15)	C33—C36A—H36A	109.5
C5—C6—C7	117.50 (14)	C33—C36A—H36B	109.5
C3—C4—C15	123.42 (14)	C33—C36A—H36C	109.5
C5—C4—C3	117.17 (14)	H36A—C36A—H36B	109.5
C5—C4—C15	119.38 (14)	H36A—C36A—H36C	109.5
C6—C5—H5	119.2	H36B—C36A—H36C	109.5
C4—C5—C6	121.57 (15)	C33—C34B—H34D	109.5
C4—C5—H5	119.2	C33—C34B—H34E	109.5
C14—C11—C2	112.03 (13)	C33—C34B—H34F	109.5
C14—C11—C13	108.30 (15)	H34D—C34B—H34E	109.5
C14—C11—C12	106.86 (14)	H34D—C34B—H34F	109.5
C13—C11—C2	109.14 (14)	H34E—C34B—H34F	109.5
C12—C11—C2	110.24 (14)	C33—C36B—H36D	109.5
C12—C11—C13	110.23 (14)	C33—C36B—H36E	109.5
C16—C15—C4	109.40 (13)	C33—C36B—H36F	109.5
C16—C15—C17	109.46 (14)	H36D—C36B—H36E	109.5
C18—C15—C4	112.18 (13)	H36D—C36B—H36F	109.5
C18—C15—C16	108.16 (13)	H36E—C36B—H36F	109.5
C18—C15—C17	108.75 (14)	C33—C35B—H35D	109.5
C17—C15—C4	108.86 (13)	C33—C35B—H35E	109.5
C6—C7—H7	118.7	C33—C35B—H35F	109.5
N1—C7—C6	122.63 (15)	H35D—C35B—H35E	109.5
N1—C7—H7	118.7	H35D—C35B—H35F	109.5
N1—C8—C9	131.15 (14)	H35E—C35B—H35F	109.5

C24—C23—C22—C21	−1.1 (2)	C3—C2—C1—O1	−178.44 (15)
C24—C23—C22—C33	178.44 (15)	C3—C2—C11—C14	−6.4 (2)
C24—C25—N3—C26	−177.95 (14)	C3—C2—C11—C13	113.56 (17)
C20—C21—C22—C23	0.2 (2)	C3—C2—C11—C12	−125.23 (17)
C20—C21—C22—C33	−179.33 (15)	C3—C4—C5—C6	0.3 (2)
C19—C24—C23—C22	0.7 (2)	C3—C4—C15—C16	132.25 (17)
C19—C24—C25—N3	−0.7 (3)	C3—C4—C15—C18	12.2 (2)
C19—C20—C21—C22	1.0 (2)	C3—C4—C15—C17	−108.18 (18)
C19—C20—C29—C32	−177.90 (15)	C1—C2—C3—C4	0.0 (2)
C19—C20—C29—C31	62.7 (2)	C1—C2—C11—C14	175.85 (15)
C19—C20—C29—C30	−59.0 (2)	C1—C2—C11—C13	−64.2 (2)
C23—C24—C19—C20	0.6 (2)	C1—C2—C11—C12	57.0 (2)
C23—C24—C19—O3	−179.18 (14)	C1—C6—C5—C4	1.1 (3)
C23—C24—C25—N3	177.16 (15)	C1—C6—C7—N1	0.1 (3)
C23—C22—C33—C34A	59.7 (2)	C6—C7—N1—C8	−178.61 (15)
C23—C22—C33—C35A	−56.2 (3)	C5—C6—C7—N1	−178.96 (16)
C23—C22—C33—C36A	−177.8 (2)	C5—C4—C15—C16	−49.8 (2)
C23—C22—C33—C34B	129.6 (3)	C5—C4—C15—C18	−169.79 (15)
C23—C22—C33—C36B	10.6 (4)	C5—C4—C15—C17	69.81 (19)
C23—C22—C33—C35B	−115.1 (3)	C11—C2—C3—C4	−177.87 (15)

C21—C20—C19—C24	−1.4 (2)	C11—C2—C1—C6	179.33 (15)
C21—C20—C19—O3	178.37 (14)	C11—C2—C1—O1	−0.6 (2)
C21—C20—C29—C32	3.7 (2)	C15—C4—C5—C6	−177.81 (15)
C21—C20—C29—C31	−115.72 (17)	C7—C6—C5—C4	−179.82 (15)
C21—C20—C29—C30	122.57 (17)	C8—N2—O2—C10	−0.86 (17)
C21—C22—C33—C34A	−120.8 (2)	C8—C9—C10—O2	−0.3 (2)
C21—C22—C33—C35A	123.3 (2)	O1—C1—C6—C5	177.88 (15)
C21—C22—C33—C36A	1.7 (3)	O1—C1—C6—C7	−1.1 (3)
C21—C22—C33—C34B	−50.9 (3)	N1—C8—N2—O2	−178.97 (13)
C21—C22—C33—C36B	−169.8 (3)	N1—C8—C9—C10	179.32 (17)
C21—C22—C33—C35B	64.5 (3)	N2—C8—N1—C7	172.51 (15)
C25—C24—C19—C20	178.43 (14)	N2—C8—C9—C10	−0.3 (2)
C25—C24—C19—O3	−1.4 (2)	N2—O2—C10—C9	0.7 (2)
C25—C24—C23—C22	−177.18 (15)	N3—C26—N4—O4	−179.42 (12)
C26—C27—C28—O4	0.20 (19)	N3—C26—C27—C28	179.46 (16)
C29—C20—C19—C24	−179.95 (14)	C9—C8—N1—C7	−7.1 (3)
C29—C20—C19—O3	−0.1 (2)	C9—C8—N2—O2	0.71 (19)
C29—C20—C21—C22	179.55 (15)	N4—C26—N3—C25	167.66 (15)
C2—C3—C4—C5	−0.9 (3)	N4—C26—C27—C28	−0.46 (19)
C2—C3—C4—C15	177.16 (15)	N4—O4—C28—C27	0.10 (19)
C2—C1—C6—C5	−2.0 (3)	C27—C26—N3—C25	−12.3 (3)
C2—C1—C6—C7	178.96 (15)	C27—C26—N4—O4	0.52 (18)
C3—C2—C1—C6	1.5 (2)	C28—O4—N4—C26	−0.38 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C23—H23···O2^{i}	0.95	2.60	3.5232 (19)	165
C25—H25···N2^{i}	0.95	2.70	3.637 (2)	169
C5—H5···O4^{ii}	0.95	2.66	3.538 (2)	155
C7—H7···N4^{ii}	0.95	2.82	3.708 (2)	156
C18—H18*B*···N2^{iii}	0.98	2.67	3.559 (2)	152
O1—H1···N1	0.92 (2)	1.76 (2)	2.6207 (18)	153 (2)
O3—H3···N3	0.91 (3)	1.77 (2)	2.6062 (17)	151 (2)
C10—H10···O3^{ii}	0.95	2.53	3.187 (2)	127
C28—H28···O1^{i}	0.95	2.69	3.3370 (19)	126

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) −*x*+1, −*y*+1, −*z*; (iii) −*x*, −*y*, −*z*.